




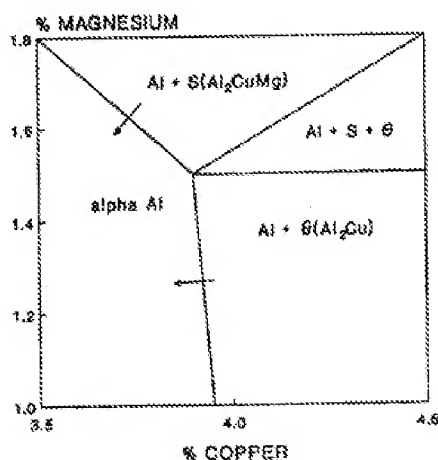


**A METHOD OF MANUFACTURING ALUMINUM AIRCRAFT SHEET****Publication number:** WO9629440**Publication date:** 1996-09-26**Inventor:** DORWARD RALPH C; KENNEDY STEPHEN D**Applicant:** KAISER ALUMINIUM CHEM CORP (US)**Classification:****- international:** C22F1/00; C22C21/12; C22C21/16; C22F1/057;  
C22F1/00; C22C21/12; C22F1/057; (IPC1-7): C22F1/04**- European:** C22C21/16; C22F1/057**Application number:** WO1996US03390 19960320**Priority number(s):** US19950407842 19950321; US19960597540 19960202**Also published as:** EP0817870 (A1) US5938867 (A1) EP0817870 (A4) EP0817870 (A0)**Cited documents:** US5213639[Report a data error here](#)**Abstract of WO9629440**

A method of producing an aluminum product comprising: providing stock including an aluminum alloy comprising about 4.0 to 4.4 wt.% copper, about 1.25 to 1.5 wt.% magnesium, about 0.35 to 0.5 wt.% manganese, not more than 0.12 wt.% silicon, not more than 0.08 wt.% iron, not more than 0.06 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities; hot working the stock; annealing; cold rolling; solution heat treating; cooling; holding for at least 12 hours at room temperature; and cold working from about 4 % to 7 % thereby producing a product having increased strength and toughness properties. The figure shows composition-phase relations for an Al-Cu-Mg system at 930 DEG F.



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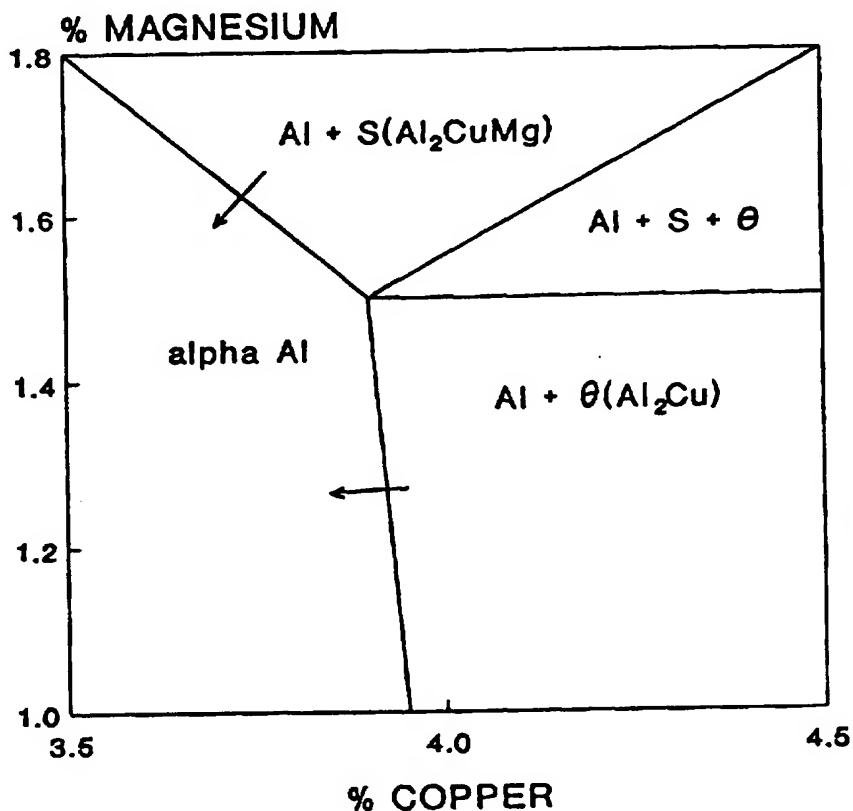
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(54) Title: A METHOD OF MANUFACTURING ALUMINUM AIRCRAFT SHEET

## (57) Abstract

A method of producing an aluminum product comprising: providing stock including an aluminum alloy comprising about 4.0 to 4.4 wt.% copper, about 1.25 to 1.5 wt.% magnesium, about 0.35 to 0.5 wt.% manganese, not more than 0.12 wt.% silicon, not more than 0.08 wt.% iron, not more than 0.06 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities; hot working the stock; annealing; cold rolling; solution heat treating; cooling; holding for at least 12 hours at room temperature; and cold working from about 4 % to 7 % thereby producing a product having increased strength and toughness properties. The figure shows composition-phase relations for an Al-Cu-Mg system at 930 °F.



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## **A Method of Manufacturing Aluminum Aircraft Sheet**

### 5      **1. Field of Invention**

          This invention relates to aluminum alloys suitable for use in aircraft applications. More specifically, it relates to a method of making an improved aluminum product having improved damage tolerant characteristics, including improved fracture toughness, fatigue  
10      resistance, corrosion resistance, formability and surface roughness properties.

### **2. Description of the Related Art**

          The design of commercial aircraft requires different sets of properties for different types of structures. Depending on the design  
15      criteria for a particular airplane component, improvements in fracture toughness and fatigue resistance result in weight savings, which translate to fuel economy over the lifetime of the aircraft, and/or a greater level of safety. For example, a slower fatigue crack growth rate will require a longer time for a crack or flaw to grow to a size where  
20      it becomes "critical" leading to catastrophic failure; and higher fracture toughness means that a crack can grow to a longer length before it is critical.

          Corrosion damage has been a perennial problem in today's aircraft, and the fuselage is the prime location for corrosion to occur.  
25      Improvements in corrosion resistance, therefore, are often sought with or without weight savings.

          The issues of toughness, fatigue and corrosion all relate to structural integrity of the airplane. Also, the aerospace manufacturers

have long established an interest in sheet products that exhibit improved formability as a means to reduce manufacturing costs. An improved formability sheet product is able to reduce the number of forming steps associated with the fabrication of a given part, in addition to avoiding the scrap associated with difficult-to-make parts.

For some time, heat treatable aluminum base alloy sheet and plate containing copper, magnesium and manganese has found considerable acceptance for various structural members. Such alloys generally contain 3.8 to 4.9 wt.% copper, 1.2 to 1.8 wt.% magnesium and 0.3 to 0.9 wt.% manganese and carries the Aluminum Association designation of 2024 alloy. This alloy is noted for its superior strength to weight ratio, its good toughness and tear resistance, and adequate resistance to general and stress corrosion effects.

Workers in the field have generally adapted the 2024 alloy for use in the construction of commercial aircraft. For example, one alloy used on the lower wing skins of some commercial jet aircraft is alloy 2024 in the T351 temper. Alloy 2024-T351 has a relatively high strength-to-density ratio and exhibits reasonably good fracture toughness, good fatigue properties, and adequate corrosion resistance. U.S. Pat. Nos. 4,336,075 to Quist et al. and 4,294,625 to Hyatt et al. disclose an alloy which has a higher strength to density ratio, improved fatigue and fracture toughness characteristics over alloy 2024 while maintaining corrosion resistance levels approximately equal to or slightly better than 2024. Quist et al. and Hyatt et al. achieve their improvements by homogenizing the alloy at a moderate temperature, carefully controlling the hot-rolling and extrusion parameters and then natural age-hardening to produce a highly elongated, substantially unrecrystallized microstructure. Similarly, U.S. Pat. No. 5,213,639 to Colvin et al. discloses an alloy which has at least a 5.0% improvement over 2024 alloy in T-L fracture toughness or fatigue crack growth rate

by re-heating the alloy prior to hot rolling. Yet no one has been able to develop an alloy which combines all of the above mentioned properties as well as significantly improving the formability of the T3 condition to impact manufacturing costs and ease of manufacturing.

5           There still remains a need, therefore, for an improved alloy that has increased fracture toughness, fatigue resistance, corrosion resistance and formability over alloy 2024, particularly in the T3 condition. Accordingly, it is the primary object of this invention to provide such an alloy.

#### 10       **Summary of the Invention**

          The present invention provides a product comprising an aluminum base alloy including about 3.8 to 4.5 wt.% copper, about 1.2 to 1.6 wt.% magnesium, about 0.3 to 0.6 wt.% manganese, not more than about 0.15 wt.% silicon, not more than about 0.12 wt.% iron, not  
15       more than about 0.1 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities, the product having at least 5% improvement over 2024 alloy in fracture toughness, fatigue crack growth rate, corrosion resistance, and formability properties.

          In an alternative embodiment, the invention provides a method  
20       of producing an aluminum product comprising providing stock including an aluminum alloy comprising about 3.8 to 4.9 wt.% copper, about 1.2 to 1.8 wt.% magnesium, about 0.3 to 0.9 wt.% manganese, not more than 0.30 wt.% silicon, not more than 0.30 wt.% iron, not more than 0.15 wt.% titanium, the remainder substantially aluminum,  
25       incidental elements and impurities; hot working the stock; annealing; cold rolling; solution heat treating; and cooling thereby producing an alloy having improved fracture toughness, fatigue resistance, corrosion resistance, and formability properties.

In another embodiment, the invention provides a method of producing an aluminum product having improved formability properties. The method includes providing stock comprising an aluminum alloy comprising about 3.8 to 4.9 wt.% copper, about 1.2 to 1.8 wt.% magnesium, about 0.3 to 0.9 wt.% manganese, not more than .30 wt.% silicon, not more than .30 wt.% iron, not more than 0.15 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities; hot working the stock; annealing; solution heat treating; cooling; and minimal cold working to produce an improved alloy having increased formability.

In a further embodiment, we provide a method of producing an aluminum product having optimized strength and toughness properties. The method includes the above process except that after the cooling step, the product is held until the alloy obtains a stable condition. The product is then cold worked to attain increased strength properties with good toughness properties.

The foregoing and other objects, features, and advantages of the invention will become more readily apparent from the following detailed description of preferred embodiment which proceeds with reference to the drawings.

#### **Brief Description of the Drawings**

FIG. 1 shows composition-phase relations for an Al-Cu-Mg system at 930°F.

FIG. 2 is a graph showing fracture toughness ( $K_{app}$ ) as a function of iron content.

FIG. 3 is a graph showing tear strength--yield strength ratio (TYR) as a function of iron content.

FIG. 4 is a graph showing fracture toughness ( $K_{app}$ ) as correlated with manganese and iron levels.

FIG. 5 is a graph showing tear strength--yield strength ratio (TYR) as correlated with manganese and iron levels.

FIG. 6 is a graph showing formability parameters as a function of iron and manganese levels.

5        FIG. 7 is a graph showing unit propagation energy of alloys having 0.54 wt.% and 0.98 wt.% Mn fabricated with and without an intermediate anneal

10        FIG. 8a is a photograph showing the improved alloy having 0.54 wt.% Mn without intermediate annealing and FIG. 8b is a photograph of the same alloy with intermediate annealing according to the present invention.

15        FIG. 9a is a photograph showing the improved alloy having 0.98 wt.% Mn without intermediate annealing and FIG. 9b is a photograph of the same alloy with intermediate annealing according to the present invention.

FIG. 10 is a graph showing yield strength as a function of total cold work after solution heat treatment, according to the present invention.

20        FIG. 11 is a graph showing ultimate strength as a function of total cold work after solution heat treatment, according to the present invention.

FIG. 12 is a graph showing yield strength as a function of total cold work after solution heat treatment, according to the present invention.

25        FIG. 13 is a graph showing ultimate strength as a function of total cold work after solution heat treatment, according to the present invention.

FIG. 14 is a graph showing elongation as a function of yield strength, according to the present invention.



FIG. 15 is a graph showing toughness as a function of yield strength, according to the present invention.

FIG. 16 is a graph showing fatigue crack growth rate as a function of cold work after solution heat treatment, according to the present invention.

FIG. 17 is a graph showing a comparison of an alloy according to the present invention to a conventional AA 2024 alloy regarding fatigue crack growth rate as a function of Delta K.

#### **Detailed Description of the Invention**

The fracture toughness, fatigue resistance, corrosion resistance, and formability properties of the present invention are dependent upon a chemical composition that is closely controlled within specific limits as set forth below and upon carefully controlled and sequenced process steps. If the composition limits or process parameters stray from the limits set forth below, the desired combination of fracture toughness, fatigue resistance, corrosion resistance, formability, and surface smoothness objectives will not be achieved.

The aluminum alloy of the present invention comprises about 3.8 to 4.5 wt.% copper, about 1.2 to 1.6 wt.% magnesium, about 0.3 to 0.6 wt.% manganese, not more than about 0.15 wt.% silicon, not more than about 0.12 wt.% iron, and not more than about 0.10 wt.% titanium, the balance being aluminum and impurity elements. For any remaining trace elements, each has a maximum limit 0.05 wt.%, with a total maximum of 0.15 wt.%. A preferred alloy would comprise about 4.0 to 4.4 wt.% copper, about 1.25 to 1.5 wt.% magnesium, about 0.35 to 0.50 wt.% manganese, not more than about 0.12 wt.% silicon, not more than about 0.08 wt.% iron, and not more than about 0.06 wt.% titanium, the balance being aluminum and impurity elements.

The chemical composition of the alloy of the present invention is similar to that of alloy 2024, but is distinctive in several important aspects. The alloying elements contained in the allowed range of variation for alloying elements contained in the invention alloy is less  
5 than for 2024. This is important because many mechanical and physical properties change as composition changes. To maintain the desired close balance of properties of the invention it is therefore necessary to restrict composition changes to a greater degree than is normally done. In addition, to the restricted ranges of copper,  
10 magnesium, and manganese, the silicon, iron, and titanium concentrations are reduced to the lowest levels commercially feasible for aluminum alloys of the present type in order to improve the fracture toughness.

#### **Improved Fracture Toughness**

15 The "damage tolerant" design philosophy being used today for commercial and military aircraft assumes that all structures contain flaws (cracks). The stress at the tip of a sharp crack is characterized by a stress intensity factor K, given by

$$K = Y\sigma\sqrt{c}.$$

20 where  $\sigma$  is the average applied stress on the structure (pounds per square inch), Y is a dimensionless parameter dependant on the geometry of the structural member, and c is the crack length. The stress intensity factor at which the crack begins to extend, generally resulting in catastrophic failure, is known as the fracture toughness of  
25 the material.

We now consider the factors that affect the fracture toughness of heat treated aluminum alloys, i.e., those alloys that derive their strength from thermal treatments such as a "solutionizing" operation that dissolves the alloying elements, followed by rapidly cooling to

room temperature (quenching), and then "aging" at room temperature or higher (250 to 375°F) to precipitate the alloying elements as small discrete particles within the aluminum matrix. Two major controls must be maintained on the chemical composition of these alloys. First, the iron impurity level must be kept to a minimum, because it is insoluble in aluminum and forms coarse, brittle intermetallic particles that contribute to crack extension and fracture. Second, the amounts of the major alloying elements, such as copper and magnesium, should be controlled to ensure that they are dissolved into the aluminum matrix during the solutionizing operation. Any intermetallic particles that are left undissolved reduce fracture toughness as do those resulting from high iron levels.

FIG. 1 graphically illustrates an equilibrium phase diagram for the aluminum (Al) - copper (Cu) - magnesium (Mg) system at 930°F. Specifically, FIG. 1 defines the copper and magnesium concentrations that can be dissolved. If the limits defined by the alpha aluminum region are exceeded, undissolved particles of  $\text{Al}_2\text{CuMg}$  (commonly designated as "S" phase) and  $\text{Al}_2\text{Cu}$  (commonly designated as " $\theta$ " phase), remain after solution heat treatment. This situation is complicated by the presence of iron, which can combine with copper to form an insoluble  $\text{Al}_7\text{Cu}_2\text{Fe}$  intermetallic constituent. The copper level in FIG. 1 therefore must be adjusted upwards by an amount equal to approximately twice the iron concentration because the  $\text{Al}_7\text{Cu}_2\text{Fe}$  constituent contains about two times as much copper as iron.

A third compositional factor is the role of sparingly soluble alloying elements such as chromium, manganese and zirconium. One or more of these alloying elements are intentionally added to aluminum to form "dispersoids," which are small intermetallic particles that are useful in controlling the crystallite, or "grain" structure of aluminum alloys. All metallic products are comprised of numerous crystallites,

or grains, which should not be allowed to grow to a large size during any of the thermal processing operations, because strength and good fracture toughness are favored by small grains. The dispersoid particles act to "pin" the grains and prevent their growth.

5           The dispersoid forming element in Al-Cu-Mg alloy 2024 is manganese in the range of 0.3 to 0.9%. Unexpectedly, we have discovered a significant effect of manganese on fracture toughness as measured by two test methods. In one method, we tested to failure 16-  
10   inch wide by 36-inch long panels with a 4-inch long through-thickness sharp crack in the orientation transverse to the rolling direction (T-L). Using an equation similar to  $K = Y\sigma\sqrt{c}$ , above, we calculated values of  $K$  (apparent) or  $K_{app}$ . It is noteworthy that  $K_{app}$  determined in this manner is only an indicator of the true fracture toughness, because the  
15   stress required to cause failure exceeded the elastic limit of the material. A much wider panel would be required to obtain the actual fracture toughness, and is beyond the capability of most test laboratories. In the second method, a 1.5-inch wide by 2.25-inch long panel with a sharp notch on one side was pulled to failure and the tear  
20   strength (maximum load divided by the cross sectional area) was measured. The tear strength divided by the yield strength as determined in a standard tensile test, commonly called the tear-yield ratio (TYR), is known to correlate with fracture toughness. Table 1 illustrates a number of production lots of 2024 alloy sheets having various iron and manganese contents which we tested for toughness by the  
25   aforementioned methods. Table 2 illustrates the results of these tests.

**TABLE 1**  
**CHEMICAL COMPOSITIONS OF PRODUCTION LOTS OF**  
**2024-T3 SHEETS**  
**(Core alloy with cladding removed)**

Alloy	% by wt <sup>a</sup>						
	Si	Fe	Cu	Mn	Mg	Ti	Zn
1	<0.1	0.035	4.31	0.33	1.37	0.02	0.02
2	<0.1	0.04	4.21	0.46	1.28	0.02	0.02
3	<0.1	0.07	3.99	0.32	1.37	0.02	0.06
4	<0.1	0.07	3.99	0.44	1.28	0.04	0.22
5	<0.1	0.17	4.21	0.39	1.44	0.03	0.03
6	<0.1	0.16	4.17	0.77	1.21	0.03	0.07
7	<0.1	0.19	4.43	0.54	1.48	0.01	0.01
<sup>a</sup> By inductively coupled plasma spectroscopy.							

**TABLE 2**  
**EFFECT OF IRON AND MANGANESE**  
**CONTENTS ON TOUGHNESS OF ALCLAD 2024-T3 SHEET<sup>a</sup>**

Alloy No.	% Fe	%Mn	T-L K <sub>app</sub> (ksi√in)	T-L TS/YS <sup>b</sup>	YS <sup>c</sup> (ksi)
1	0.035	0.33	89	1.76	45.5
2	0.04	0.46	87	1.70	46.3
3	0.07	0.32	85	1.65	45.4
4	0.07	0.44	82	1.59	44.4
5	0.17	0.39	83.5	1.60	43.4
6	0.16	0.77	77.5	1.52	44.5
7	0.19	0.54	79.5	1.53	44.2
<sup>a</sup> Also: 4.0-4.5% Cu and 1.2-1.5% Mg; all 0.063" thick. <sup>b</sup> Tear strength - yield strength ratio (TYR). <sup>c</sup> Transverse tensile yield strength.					

We used data from Table 2 to plot the toughness measurements as a function of wt.% iron in FIGs. 2 and 3. As expected, FIGs. 2 and

3 demonstrate a correlation of fracture toughness with decreasing concentrations of iron. Surprisingly, however, the lots with relatively low manganese levels exhibit higher toughness values for a given iron content. Table 3, which compares the toughness levels at two manganese levels for a number of iron concentrations, also demonstrates this phenomenon. Table 3 also lists copper contents for each alloy, because high levels of copper can reduce toughness by the presence of undissolved  $Al_2Cu$  and  $Al_2CuMg$  phases. Notably, the copper levels of the alloys being compared in each case are almost equivalent.

**TABLE 3**  
**EFFECT OF MANGANESE AT VARIOUS IRON LEVELS ON**  
**TOUGHNESS**  
**OF ALCLAD 2024-T3 SHEET**

ALLOY	%MN	%CU	T-L $K_{app}$	T-L TS/YS <sup>b</sup>
0.035 - 0.04% Fe				
1	0.33	4.31	89	1.76
2	0.46	4.21	87	1.70
	$\Delta(\%)$		2.3	3.50
0.07% Fe				
3	0.32	3.99	85	1.65
4	0.44	3.99	82	1.59
	$\Delta (\%)$		3.5	3.80
0.16-0.17% Fe				
5	0.39	4.17	83.5	1.6
6	0.77	4.21	77.5	1.52
	$\Delta (\%)$		7.7	5.3
	Ave. $\Delta$ /0.1% Mn		2.2	2.4
* Tear strength - yield strength ratio (TYR).				

A linear regression analysis of the  $K_{app}$  data showed that manganese has approximately half the detrimental effect that iron has. This discovery is particularly important because of the relatively high levels of manganese in alloys such as 2024. Specifically, FIG. 4 demonstrates toughness,  $K_{app}$ , as a function of iron and manganese concentrations, producing the correlation:

$$K_{app} = 93.2 - 29.2 (\%Fe + 0.50 \%Mn)$$

Similarly, toughness, expressed as tear-yield ratio or TYR, is represented by the correlation:

$$TYR = 1.81 - 0.54(\%Fe + 0.50\%Mn)$$

This correlation is illustrated in FIG. 5.

#### **Improved Fatigue Resistance**

As noted in the previous section, the "damage tolerant" design philosophy assumes that flaws (cracks) are present in all structural materials. If these cracks are permitted to grow to a "critical" size such that the stress intensity factor at the crack tip exceeds the fracture toughness of the material, catastrophic failure occurs. Cracks can grow as a result of cyclic loads (fatigue) caused by takeoff and landing or cabin pressurization and depressurization. Fatigue crack growth rates for the projected cyclic loading stresses are therefore desirably low.

Experimentally, we found that the velocity of cracks growing under fatigue conditions, i.e., the fatigue crack growth rate, is dependent on the stress intensity factor difference ( $\Delta K$ ) associated with the minimum and maximum load. The stress intensity factor increment ( $\Delta K$ ) must therefore, be specified when comparing fatigue crack growth rates for different materials

In addition to improved toughness, we also discovered that higher purity alloys with relatively low manganese levels also had low

fatigue crack growth rates. We determined this by running tests at a stress intensity increment ( $\Delta K$ ) of 30 ksi $\sqrt{\text{in.}}$  and a load ratio (maximum load divided by minimum load) of 0.1 on several of the alloys listed in Tables 1-3. For example, alloys 1 and 2 had average crack growth rates of  $7.0 \times 10^{-5}$  and  $7.5 \times 10^{-5}$  inches/cycle, compared to a nominal value of  $20 \times 10^{-5}$  inch/cycle for standard 2024 alloy typified by alloy 7. Thus, the alloy of our invention has about a 50% decrease in crack growth rate over standard 2024 alloy at a  $\Delta K$  of 30 ksi $\sqrt{\text{in.}}$  Similarly, we discovered fatigue benefits at lower values of  $\Delta K$ . For example, at a  $\Delta K$  of 5 ksi $\sqrt{\text{in.}}$ , alloys 1,2,3, and 4 had crack growth rates of  $1.5$  to  $2.2 \times 10^{-7}$  inches/cycle compared to  $1.7$  to  $4.0 \times 10^{-7}$  inches/cycle for standard 2024 alloy. Or stated another way, our new alloy had about a 25% decrease in crack growth rate in the low  $\Delta K$  regime.

#### **Improved Corrosion Resistance**

Yet another benefit of the new alloy of my invention is improved corrosion resistance. As we noted earlier, good corrosion resistance is of prime concern in aircraft fuselage structures. Corrosion of aluminum alloys is usually aggravated by salt (sodium chloride) containing environments such as can be present near oceans. Sheet samples from alloys 3 and 7 (of Tables 1-3) were therefore exposed to a marine atmosphere at Daytona Beach, Florida for one year. The protective cladding was removed from one surface so that the inherent corrosion resistance of the core alloy could be assessed. This also simulates the practical situation where one side of a fuselage panel is chemically milled to a thinner section size. After the one-year exposure period, tensile specimens were machined from the samples, and as recommended in the Corrosion Handbook (edited by H. H. Uhlig, John Wiley & Sons, p. 956), the corrosion damage was



quantified by loss in ductility. This method is particularly suited to materials that are susceptible to pitting and intergranular corrosion. Table 4 summarizes tensile elongation measurements before and after the exposure to the marine atmosphere. Metallographic examination revealed that ductility loss corresponded with the depth of pitting corrosion attack on the exposed and corroded alloys. It is apparent that alloy 3, which has lower iron and manganese contents, is superior in corrosion resistance.

**TABLE 4**  
**EFFECT OF MARINE EXPOSURE ON DUCTILITY LOSS**

Alloy	Elongation, % in 1 inch		%Loss in Ductility
	Before	After	
3	23.5	19.1	19
7	22.5	14.5	36

**Improved Formability**

Another advantage of our invention is improved formability. Good formability is important to the aircraft manufacturers because of lower costs associated with reduced scrap rates and manpower requirements. Two indicators of formability are (1) ball punch depth as determined by indenting the sheet with a 1-inch diameter steel ball until it cracks (also known as Olsen cup depth), a measure of a material's capability of being stretched in more than one direction, and (2) minimum bend radius, a measure of a material's ability to be bent without cracking. Note that there is some uncertainty in minimum bend radius measurements because the determination of surface cracking is somewhat subjective, and the method involves bending sheet samples around dies of incremental (not continuously varying) radii. Table 5 lists minimum bend radius and ball punch depth of alloys 1, 2, 4, 6 and 7. As FIG. 6 illustrates, both of these indicators

15

correlate with % Fe + 1/2% Mn, i.e., alloys with less than about 0.1% Fe and less than about 0.5% Mn have superior formability.

**TABLE 5**  
**FORMABILITY OF 2024-T3 SHEET**

5	<b>Alloy</b>	<b>% Fe</b>	<b>% Mn</b>	<b>Olsen Cup Depth, in.</b>	<b>180°Min. Bend Radius, in.</b>
	1	0.035	0.33	0.336	0.025-0.032
	2	0.04	0.46	0.319	0.025-0.032
	4	0.07	0.44	0.333	0.032-0.064
	6	0.16	0.77	0.287	0.080-0.100
10	7	0.19	0.54	0.309	0.080-0.100

**Improved Surface Roughness**

Three lots each of standard 2024 and the invention composition were chemically milled to half thickness in a buffered 14% NaOH solution. The roughness of the milled surfaces was measured in a direction perpendicular to the rolling direction using a profilometer with a 2  $\mu$ m (2 x 10<sup>-6</sup> meters) diamond stylus. The results listed in Table 6 show a 10 to 45% improvement for the invention product.

**TABLE 6**  
**SURFACE ROUGHNESS OF CHEMICALLY MILLED SHEET**

20	<b>Gage, in.</b>	<b>Alloy</b>	<b>Roughness (X10<sup>-4</sup>in.)</b>
	0.125	IP* 2024	58 107
	0.160	IP 2024	107 119
	0.190	IP 2024	139 186
25	* Invention Product		

**Improved Physical Properties by Intermediate Annealing Step**

We have also discovered that we can further improve the properties that were discussed above by an intermediate thermal treatment. Specifically, we introduce an intermediate annealing step  
5 after hot rolling but before cold rolling to the final gage to produce an improved alloy.

For purposes of the present invention, we prefer a method which includes providing stock comprising an aluminum alloy having about 3.8 to 4.5 wt.% copper, about 1.2 to 1.6 wt.% magnesium, about 0.3  
10 to 0.6 wt.% manganese, not more than .15 wt.% silicon, not more than .12 wt.% iron, not more than 0.1 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities; hot working the stock; annealing; cold rolling; solution heat treating; and cooling.

15 Optionally, before the hot working step, we homogenize the stock to produce a substantially uniform distribution of alloying elements. In general, we homogenize by heating the stock to a temperature ranging from about 900 to 975°F for a period of at least 1.0 hour to dissolve soluble elements and to homogenize the internal structure of  
20 the metal. We caution, however, that temperatures above 935°F are likely to damage the metal and thus we avoid these increased temperatures if possible. Generally, we homogenize for at least 4.0 hours in the homogenization temperature range. Most preferably, we homogenize for about 6.0 to 12.0 hours at about 920°F.

25 As discussed above, our preferred aluminum alloy comprises about 4.0 to 4.4 wt.% copper, about 1.25 to 1.5 wt.% magnesium, about 0.35 to 0.5 wt.% manganese, not more than 0.12 wt.% silicon, not more than 0.08 wt.% iron, not more than 0.06 wt.% titanium, the remainder substantially aluminum, incidental elements and impurities.

For hot working, we prefer a hot rolling step where the stock is heated to a temperature ranging from about 750 to 925°F for about 1.0 to 12.0 hours. Most preferably, we heat the stock to a temperature ranging from about 825 to 900°F for about 1.0 to 2.0 hours to obtain a gage thickness ranging from about 0.1 to 0.25 inches. We generally perform hot rolling at a starting temperature ranging from about 600 to 900°F, or even higher as long as no melting or other ingot damage occurs. When the alloy is to be used for fuselage skins, for example, we typically perform hot rolling on ingot or starting stock 12 to 16 or more inches thick to provide an intermediate product having a thickness ranging from about 0.1 to 0.25 inches.

After hot rolling, we next anneal the stock. Preferably, we anneal at a temperature ranging from about 725 to 875°F for about 1.0 to 12.0 hours. Most preferably, we anneal the stock at a temperature ranging from about 750 to 850°F for about 4.0 to 6.0 hours at heating rate ranging from about 25 to 100°F per hour, with the optimum being about 50°F per hour.

After annealing, we next cold roll the intermediate gage stock. Preferably, we allow the annealed stock to cool to less than 100°F and most preferably to room temperature before we begin cold rolling. Preferably, we cold roll to obtain at least a 40% reduction in sheet thickness, most preferably we cold roll to a thickness ranging from about 50 to 70% of the hot rolled gage.

After cold rolling, we next solution heat treat the stock. Preferably, we solution heat treat at a temperature ranging from about 900 to about 940°F for about 10 to 30 minutes. It is important to rapidly heat the stock, preferably at a heating rate of about 100 to 2000 °F per minute. Most preferably, we solution heat treat at about 920 to 930°F for about 15 minutes at a heating rate of about 1000°F per minute.

If the temperature is substantially below 920°F, then the soluble elements, copper and magnesium are not taken into solid solution. This circumstance can be illustrated by reference to Figure 1. As the temperature is decreased, the lines encompassing the aluminum solid solution region shift to the left as depicted by the arrows. When  
5 copper and magnesium are not taken into solution, two undesirable consequences result: (1) there are insufficient alloying elements to provide adequate strength upon subsequent age hardening; and (2) the copper and magnesium-containing intermetallic compounds ( $\text{Al}_2\text{Cu}$  and  
10  $\text{Al}_2\text{CuMg}$ ) that remain undissolved detract from fracture toughness and fatigue resistance. Similarly, if the time at the solution heat treatment temperature is too short, these intermetallic compounds do not have time to dissolve. The heating rate to the solutionizing temperature is important because relatively fast rates generate a fine grain (crystallite)  
15 size, which is desirable for good fracture toughness and high strength.

After solution heat treatment, we rapidly cool the stock to minimize uncontrolled precipitation of secondary phases, such as  $\text{Al}_2\text{CuMg}$  and  $\text{Al}_2\text{Cu}$ . Preferably, we quench at a rate of about 1000°F/sec. over the temperature range 750 to 550° from the solution  
20 temperature to a temperature of 100°F or lower. Most preferably, we quench using a high pressure water spray at room temperature or by immersion into a water bath at room temperature, generally ranging from about 60 to 80°F.

### EXAMPLE 1

25 To demonstrate the present invention, we first homogenized two 3" x 9" ingots having the composition listed in Table 7 at a temperature of about 910°F for about 15 hours.

**TABLE 7**  
**CHEMICAL COMPOSITIONS OF LABORATORY INGOTS**

	% by wt.					
Alloy	Si	Fe	Cu	Mn	Mg	Ti
A	0.07	0.07	3.84	0.54	1.24	0.02
B	0.07	0.09	3.83	0.98	1.22	0.02

We then reheated the ingots to a temperature of about 800°F and hot rolled them to an intermediate gage thickness of about 0.200" having a final temperature of about 550°F. We then divided each hot rolled sheet into two sheets. We annealed one of the sheets at a temperature of about 835°F for about 2.0 hours using heating and cooling rates of about 50°F/hr. The other control sheet was not annealed. Then, we cold rolled all four sheets to a gage of about 0.063" and solution heated treated them at about 920°F for about 30 minutes. Finally, we quenched all four sheets in room temperature water. We then tested all four sheets after naturally aging them at room temperature for greater than one month (T4 temper) for tensile properties and Kahn tear energy, which are listed in Table 8.

**TABLE 8**  
**PROPERTIES WITH AND WITHOUT INTERMEDIATE ANNEAL**

Alloy	Anneal	UTS,ksi	YS,ksi	Elong, %	UPE,in-lb/in <sup>2</sup>
A	Yes	68.3	42.6	23.5	845
A	No	66.4	40.8	24.5	755
B	Yes	68.9	42.3	22	705
B	No	68.4	41.2	21	650

We used the data from Table 8 to plot yield strength versus unit propagation energy in FIG. 7. Surprisingly, the intermediate-annealed variants of both alloys were not only somewhat stronger,

they were also significantly tougher than their un-annealed counterparts. The lower manganese Alloy A also had higher toughness values than the high manganese Alloy B, as expected based on FIGS. 4 and 5 and our previous discussion, above.

5 In addition to improvements in mechanical properties, the sheets produced with intermediate anneal had improved formability as evidenced by deeper ball punch depths shown in Table 9.

10 **TABLE 9**  
**BALL PUNCH DEPTHS**  
**WITH AND WITHOUT INTERMEDIATE ANNEAL**

Alloy	Anneal	Olsen Cup Depth, in.
A	Yes	0.330
A	No	0.304
B	Yes	0.295
B	No	0.265

15 Notably, the lower manganese alloy also had superior forming behavior as would be expected based on my previous discussion.

20 In addition, when we examined the grain structures of Alloys A and B, we discovered considerably finer grain sizes in the intermediate-annealed alloys. FIGS. 8a and 9a compared to FIGS. 8b and 9b, respectively, illustrate the phenomenon of finer grain size that we observed.

#### **Improved Formability Properties by Selective Cold Working**

25 We have also discovered that we can further improve the properties of our new alloy by selective and careful use of cold working. Conventional 2000 series alloys are often cold worked after solution heat treatment to increase strength. Cold work is,

however, detrimental to formability and fracture toughness properties. For a highly desirable combination of strength and formability, we provide the invention alloy and then hot work, intermediate anneal, solution heat treat, quench and minimally cold work the product. In general, the minimal cold work includes a small amount of stretching, leveling, straightening or combinations thereof. Typically, we cold work less than 5% and preferably we use a minimized stretch of 0.5% with minimized or no leveling to achieve T3 property minimums with significantly improved formability.

## EXAMPLE 2

To demonstrate the improvement of the new alloy in combination with selective cold working, we prepared several production lots of 2024 alloy sheet. Tables 10 illustrates the chemistry of these lots.

**TABLE 10**  
**CHEMICAL COMPOSITIONS OF**  
**PRODUCTION LOTS OF 2024-T3 SHEETS**

Alloy No.	% by wt <sup>a</sup>						
	Si	Fe	Cu	Mn	Mg	Ti	Zn
1	0.04	0.08	4.01	0.36	1.32	0.010	0.01
2	0.04	0.09	3.90	0.37	1.30	0.010	0.02
3	0.04	0.09	3.98	0.36	1.32	0.009	0.02
4	0.04	0.07	4.00	0.38	1.33	0.009	0.01

<sup>a</sup> Measured by Quantometer

In processing these lots to finished sheet, we tightly controlled the amount of cold work. Unconventionally, no stretch was imparted. As we mentioned previously, this is not typical of standard 2000 series product. This sheet product was leveled only,



and leveling was strictly controlled to impart only 0.5% to 1% maximum cold work.

Those skilled in the art, will appreciate that minimum bend radius for a given gauge is an excellent measurement of material behavior. This procedure is an accurate representation of break forming of straight flange bends. Break forming and bending is extensively used in aerospace to make production parts. Therefore, this is an area where this invention offers significant manufacturing benefits and opportunities for manufacturing cost improvement.

Tables 11A and 11B illustrate the significant improvement of the invention product over conventionally produced 2024-T3. Surprisingly, the data indicate that the invention is essentially equivalent to 2024-O in bending properties.

**TABLE 11A**  
**BEND RADIUS (INCHES) OF CONVENTIONAL SHEET**

	<b>Thickness (inches)</b>	<b>2024-T3</b>	<b>2024-O</b>
5	0.04	0.16	0.06
	0.04	0.16	0.06
	0.04	0.16	0.06
	0.04	0.16	0.06
10	0.08	0.34	0.16
	0.08	0.34	0.16
	0.08	0.34	0.16
	0.08	0.34	0.16
15	0.10	0.44	0.22
	0.10	0.44	0.22
	0.10	0.44	0.22
	0.10	0.44	0.22
20	0.125	0.56	0.25
	0.125	0.56	0.25
	0.125	0.56	0.25
	0.125	0.56	0.25

**TABLE 11B**  
**BEND RADIUS OF INVENTION 2024-T3 SHEET**

	<b>Alloy No.</b>	<b>Thk. (inches)</b>	<b>Bend Dir.</b>	<b>Failure</b>	<b>Radius (inches)</b>
5	1	0.04	L	No	0.06
	1	0.04	L	No	0.06
	1	0.04	T	No	0.06
	1	0.04	T	No	0.06
	2	0.08	L	No	0.16
10	2	0.08	L	No	0.16
	2	0.08	T	No	0.16
	2	0.08	T	No	0.16
	3	0.1	L	No	0.16
	3	0.1	L	No	0.16
15	3	0.1	T	No	0.16
	3	0.1	T	No	0.16
	4	0.125	L	No	0.19
	4	0.125	L	No	0.19
	4	0.125	T	Yes	0.19
20	4	0.125	T	Yes	0.19

As discussed earlier, in addition to formability properties, we must also consider the other mechanical properties of the product. Table 12 illustrates the success of this invention in realizing acceptable properties. Importantly, the ultimate strength, yield strength, and elongation properties are well above aerospace established specification requirements for 2024-T3. Also, with respect to a 100 lot production average of conventional 2024-T3, the data reveal acceptable correlation with ultimate strength and yield strength, and superior elongation.

**TABLE 12**  
**TRANSVERSE PROPERTIES OF PRODUCTION LOTS OF**  
**BARE 2024-T3**  
**SHEET WITH CONTROLLED COLD WORK IMPARTED BY**  
**LEVELING**

5

10

15

20

Alloy No. (Gauge)	UTS, ksi	YS, ksi	Elong, %
1 (0.040")	67.8	46.3	21.5
1 (0.040")	67.8	46.3	21.0
2 (0.080")	66.0	44.8	23.9
2 (0.080")	66.2	45.4	23.3
3 (0.100")	65.1	43.3	24.4
3 (0.100")	65.6	43.3	23.7
4 (0.125")	66.9	44.7	23.9
4 (0.125")	66.9	44.7	24.1
2024-T3 Minimum Specification Requirement (0.010"-0.128")	63.0	42.0	15
Average 2024-T3 Values (100 Lots)	68.3	46.9	18.5

**Improved Combination of Strength and Toughness by Selective Cold Working**

25

30

In addition, we have discovered that by employing a holding step prior to cold working, we can further enhance the strength and toughness properties of the invention alloy. This is important because aircraft manufacturers have an interest in fuselage sheet and light gage plate products with higher strengths than 2024-T3. Although 2024-T361 has higher strength than 2024-T3, it has not been considered for many applications because its toughness is lower than that of 2024-T3.

35

Thus, in an alternative embodiment of the invention, we describe a method for producing a T36 temper product which has an improved combination of strength and toughness. In general, we use

our preferred chemistry, hot work, anneal, solution heat treat and quench steps. Next, we hold the sheet until it reaches a stable condition. As used herein, we define "stable condition" to be such that the product has achieved 95% of its inherent strength level, thereby experiencing little further increase in strength with increasing natural aging time at room temperature. Typically, we hold the product for at least 12 hours but generally not longer than two weeks. After we achieve a stable condition, we then cold work the sheet to impart a T36 temper. This embodiment of our invention is illustrated in Example 3.

### EXAMPLE 3

We sheared fourteen 8" wide by 24" long panels from a 0.063" production lot of Alclad sheet having the composition within the preferred range of the invention chemistry as set forth in Table 13:

**TABLE 13**  
**CHEMICAL COMPOSITION OF SPECIAL CHEMISTRY 2024 SHEET**

% by wt. <sup>a</sup>						
Si	Fe	Cu	Mn	Mg	Zn	Ti
0.05	0.07	4.05	0.45	1.27	0.23	0.05
<sup>a</sup> Through-thickness composition of core alloy by ICP analysis except for Si (Quantometer melt analysis).						

We solution heat treated the panels at 920°F for 15 min. and quenched them in room temperature water. Table 13 sets forth the various cold roll/stretch combinations that we applied -- note that some sheets were cold worked immediately (<1 hr.) after quenching; others were strained after a 4-day delay.

**TABLE 14**  
**COLD WORK SCHEDULE APPLIED**  
**TO SPECIAL CHEMISTRY 2024 SHEET**

	Delay	% Cold Roll	% Stretch	Total Strain, %
5	--	0	0	0
	0	0	4	4.0
	0	6.1	2	8.1
	0	5.3	1*	6.3
	0	4.7	1*	5.7
10	0	8.5	1*	9.5
	0	5.1	2*	7.1
	0	5.1	4*	9.1
	4d	0	1	1.0
	4d	0	2	2.0
15	4d	0	4	4.0
	4d	0	8	8.0
	4d	3.2	2	5.2
	4d	3.7	1	4.7
* Delay between cold rolling and stretching was 4 days.				

20           After about two weeks of natural aging, we tested the panels for longitudinal and transverse tensile properties and T-L Kahn tear properties (toughness indicators). We also tested selected samples for T-L fatigue crack growth rate at a stress ratio of 0.1.

25           The yield and tensile strengths are plotted against % strain in Figures 10 through 13. The data separate into two trend lines: one for the 4-day delay between solution heat treating and cold work; the other for no delay. The 4-day delay gave substantially higher strengths for a given level of cold work, requiring about 4% strain to

achieve the 48 ksi minimum T361 yield strength. Without a delay, achieving the minimum yield strength required about 7% cold work. The minimum transverse ultimate strength was easier to meet (Figure 11): 0% cold work with no delay, 4% with a delay. Notably, when a  
5 2-4% stretch superseded immediate cold rolling, the strengths fell on the "No-Delay" curve, even if there was a 4-day delay between the two operations. This shows that immediate cold work must be minimized.

Figure 14, a correlation plot between transverse elongation and  
10 strength, shows that a better combination of properties was achieved with the 4-day delay. All the elongation data were comfortably above the 9% minimum for 2024-T361.

The Kahn tear unit propagation energies (UPE) are plotted against transverse yield strength in Figure 15. As with elongation, a  
15 better combination of UPE and strength was achieved with the 4-day delay. According to Figure 15, sheet with a yield strength of 51-53 ksi, should have a UPE of about 500 in.-lb./in.<sup>2</sup>, approximately the same as conventional 2024-T3 with a yield strength of only about 45 ksi. Of course, depending on the aircraft design requirements, the  
20 combination of strength and toughness values can be adjusted by varying the amount of cold work.

Six samples representing delay times of 0 and 4 days with 0 to 9.5% strain were tested for fatigue crack growth rate (FCGR) together with six conventional 2024 sheet samples given identical  
25 mechanical treatments. Figure 16, a combination of the data for the two materials, shows that FCGR is independent of cold work over the  $\Delta K$  range of 5 to 15 ksi  $\sqrt{\text{in}}$ . The averaged  $da/dN$  data for each material are compared in Figure 17, which shows lower crack growth rates for the invention composition by an average of about 10-35%.

Having illustrated and described the principles of our invention in a preferred embodiment thereof, it should be readily apparent to those skilled in the art that the invention can be modified in arrangement and detail without departing from such principles. We  
5 claim all modifications coming within the spirit and scope of the accompanying claims.



## CLAIMS

We claim:

1. A method of producing an aluminum product comprising:
  - (a) providing stock comprising an aluminum alloy comprising  
5 about 3.8 to 4.9 wt. % copper, about 1.2 to 1.8 wt. % magnesium,  
about 0.3 to 0.9 wt. % manganese, not more than .30 wt. % silicon,  
not more than .30 wt. % iron, not more than 0.15 wt. % titanium, the  
remainder substantially aluminum, incidental elements and impurities;
  - (b) hot working the stock;
  - 10 (c) annealing;
  - (d) solution heat treating;
  - (e) cooling; and
  - (f) minimal cold working to produce an improved alloy having  
increased formability.
- 15 2. The method of claim 1 wherein the cold working is selected  
from the group consisting of stretching, straightening, leveling and  
combinations thereof.
3. The method of claim 2 wherein the cold work comprises less  
than 2.0% stretching.
- 20 4. The method of claim 1 wherein the alloy of step (a)  
comprises about 4.0 to 4.4 wt. % copper, about 1.25 to 1.5 wt. %  
magnesium, about 0.35 to 0.5 wt. % manganese, not more than .12  
wt. % silicon, not more than .08 wt. % iron, not more than 0.06  
wt. % titanium, the remainder substantially aluminum, incidental  
25 elements and impurities.

5. The method of claim 1 wherein step (b) comprises hot rolling the stock at a temperature ranging from about 750 to 925°F for about 1 to 12 hours to obtain a gage thickness ranging from about 0.1 to .25 inches.

5           6. The method of claim 1 wherein step (c) comprises annealing at a temperature ranging from about 725 to 875°F for about 1 to 12 hours.

7. The method of claim 1 wherein step (d) comprises solution heat treating at a temperature ranging from about 900 to 940°F for  
10           about 10 to 30 minutes.

8. The method of claim 1 wherein step (e) comprises cooling by quenching.

9. The method of claim 1 further comprising cold rolling after step (c) annealing.

15           10. A method of producing an aluminum product comprising:  
            (a) providing stock comprising an aluminum alloy comprising about 3.8 to 4.9 wt. % copper, about 1.2 to 1.8 wt. % magnesium, about 0.3 to 0.9 wt. % manganese, not more than .30 wt. % silicon, not more than .30 wt. % iron, not more than 0.15 wt. % titanium, the  
20           remainder substantially aluminum, incidental elements and impurities;  
            (b) hot working the stock;  
            (c) annealing;  
            (d) solution heat treating;  
            (e) cooling;  
25           (f) holding the sheet to obtain a stable condition; and

(g) cold working to produce an improved alloy having increased strength and toughness properties.

11. The method of claim 10 wherein step (f) comprises holding for at least 12 hours at room temperature.

5           12. The method of claim 10 wherein the cold working is selected from the group consisting of stretching, straightening, leveling and combinations thereof.

13. The method of claim 10 wherein the amount of cold work is sufficient to impart a T36 temper.

10           14. The method of claim 10 wherein the amount of cold work ranges from about 4% to 7%.

15           15. The method of claim 10 wherein the alloy of step (a) comprises about 4.0 to 4.4 wt. % copper, about 1.25 to 1.5 wt. % magnesium, about 0.35 to 0.5 wt. % manganese, not more than .12 wt. % silicon, not more than .08 wt. % iron, not more than 0.06 wt. % titanium, the remainder substantially aluminum, incidental elements and impurities.

20           16. The method of claim 10 wherein step (b) comprises hot rolling the stock at a temperature ranging from about 750 to 925°F for about 1 to 12 hours to obtain a gage thickness ranging from about 0.1 to .25 inches.

17. The method of claim 10 wherein step (c) comprises annealing at a temperature ranging from about 725 to 875°F for about 1 to 12 hours.

5 18. The method of claim 10 wherein step (d) comprises solution heat treating at a temperature ranging from about 900 to 940°F for about 10 to 30 minutes.

19. A product produced by the method of claim 1.

20. A product produced by the method of claim 10.

21. A method of producing an aluminum product comprising:  
10 (a) providing stock comprising an aluminum alloy comprising about 4.0 to 4.4 wt. % copper, about 1.25 to 1.5 wt. % magnesium, about 0.35 to 0.5 wt. % manganese, not more than .12 wt. % silicon, not more than .08 wt. % iron, not more than 0.06 wt. % titanium, the remainder substantially aluminum, incidental elements and impurities;  
15 (b) hot working the stock;  
(c) annealing;  
(d) solution heat treating;  
(e) cooling;  
(f) holding the sheet for at least 12 hours; and  
20 (g) cold working from about 4% to 7% to produce an improved alloy having increased strength and toughness properties.

22. A product produced by the method of claim 21.

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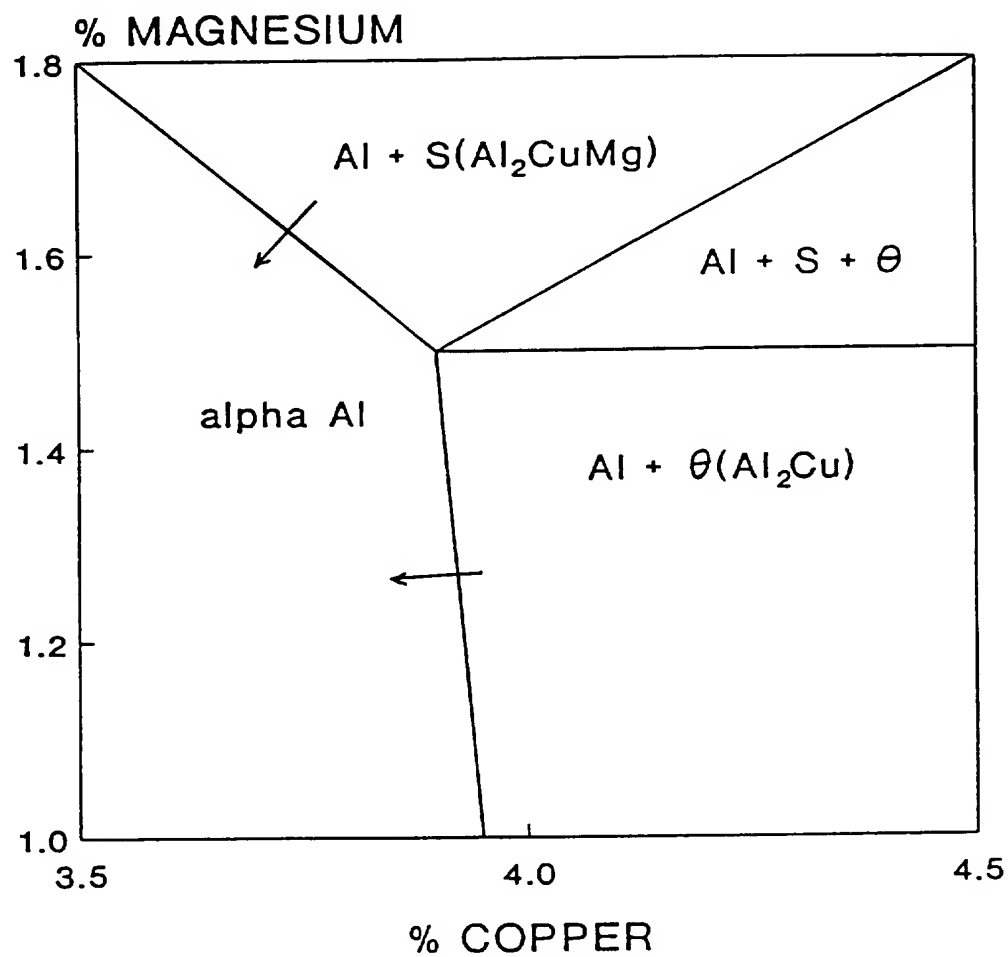


FIG. 1

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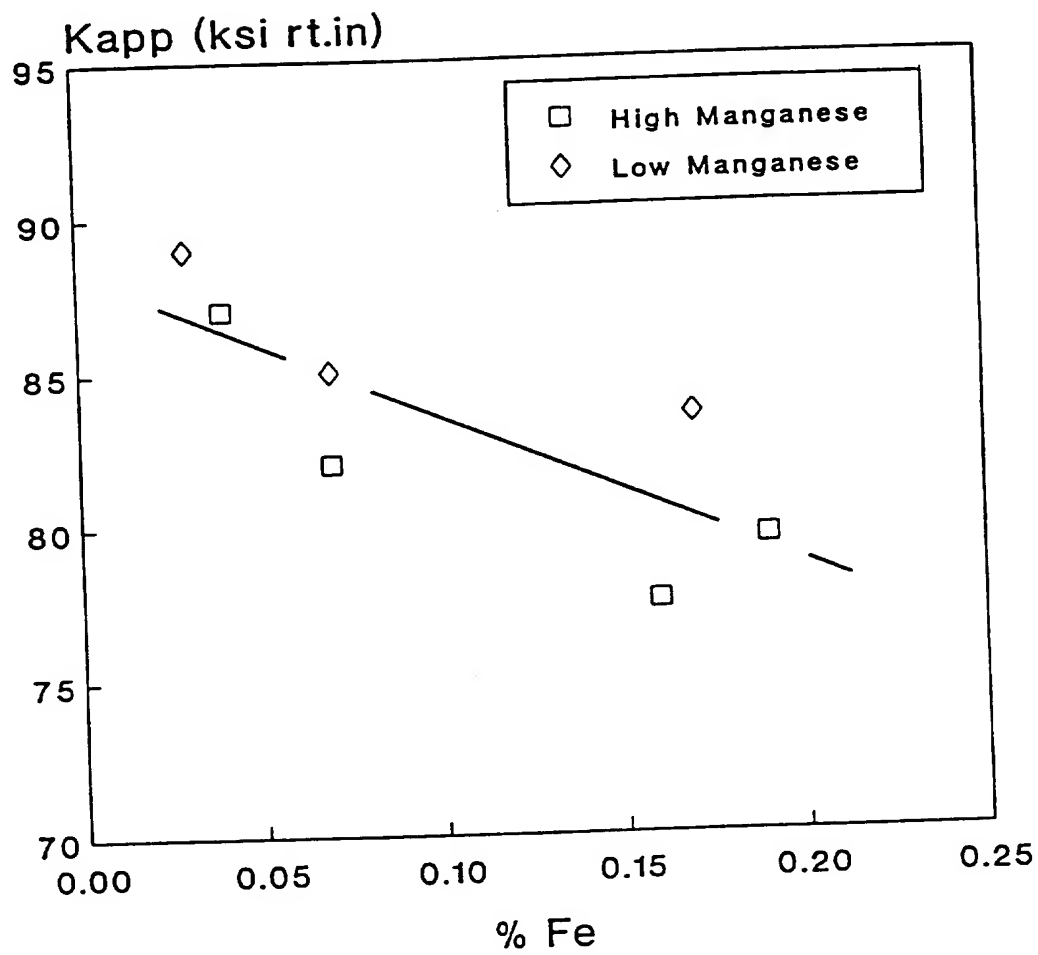


FIG. 2

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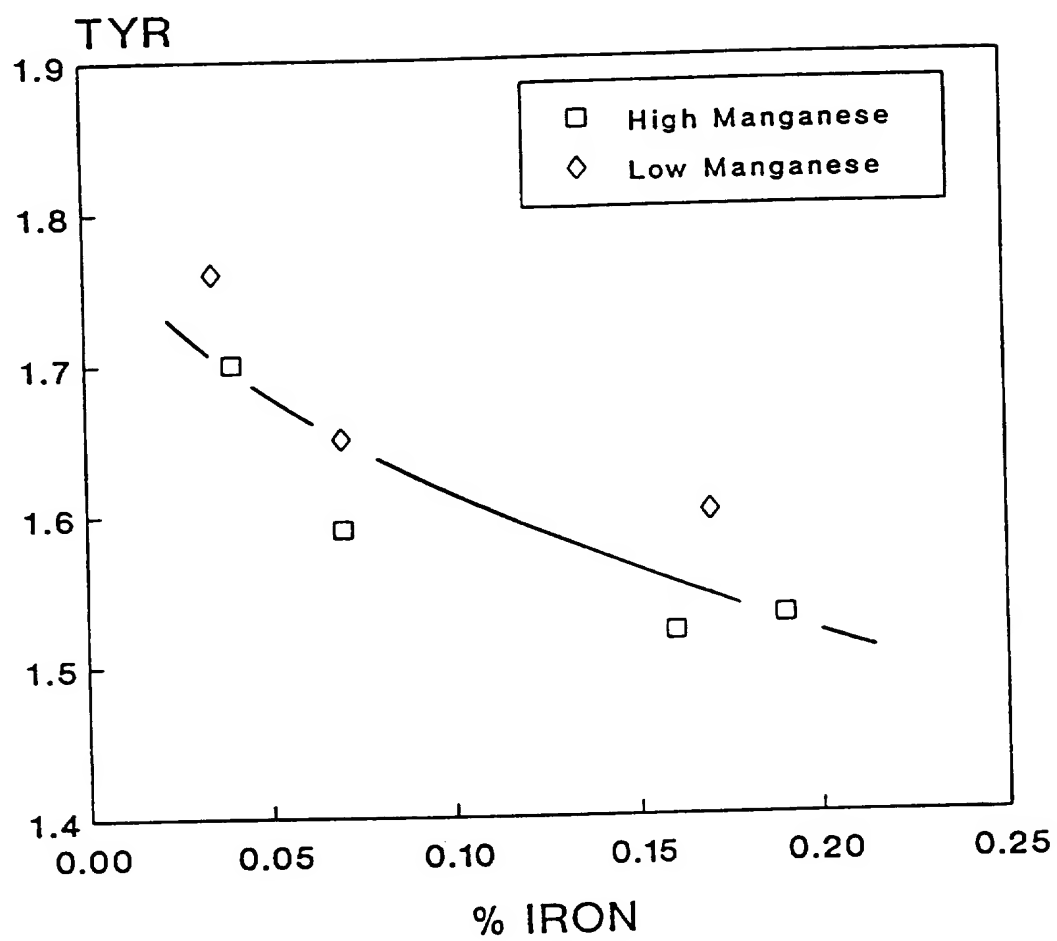


FIG. 3

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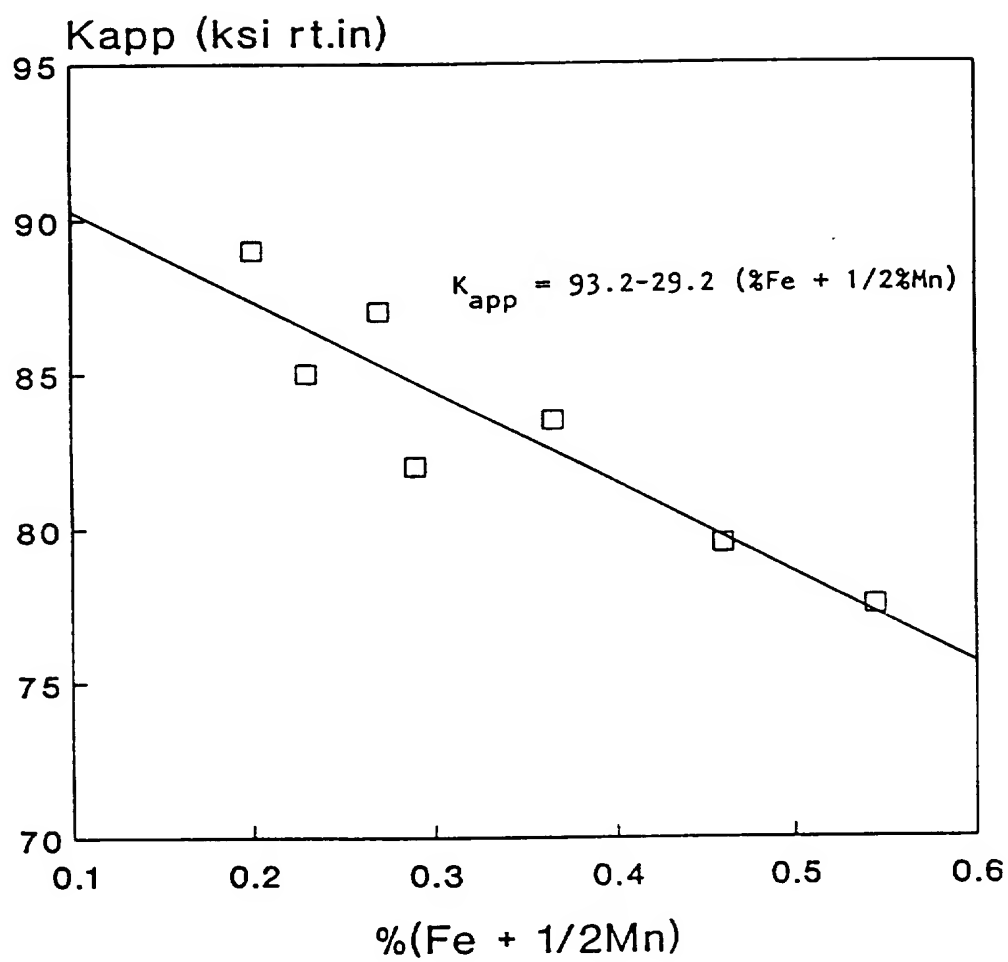


FIG. 4



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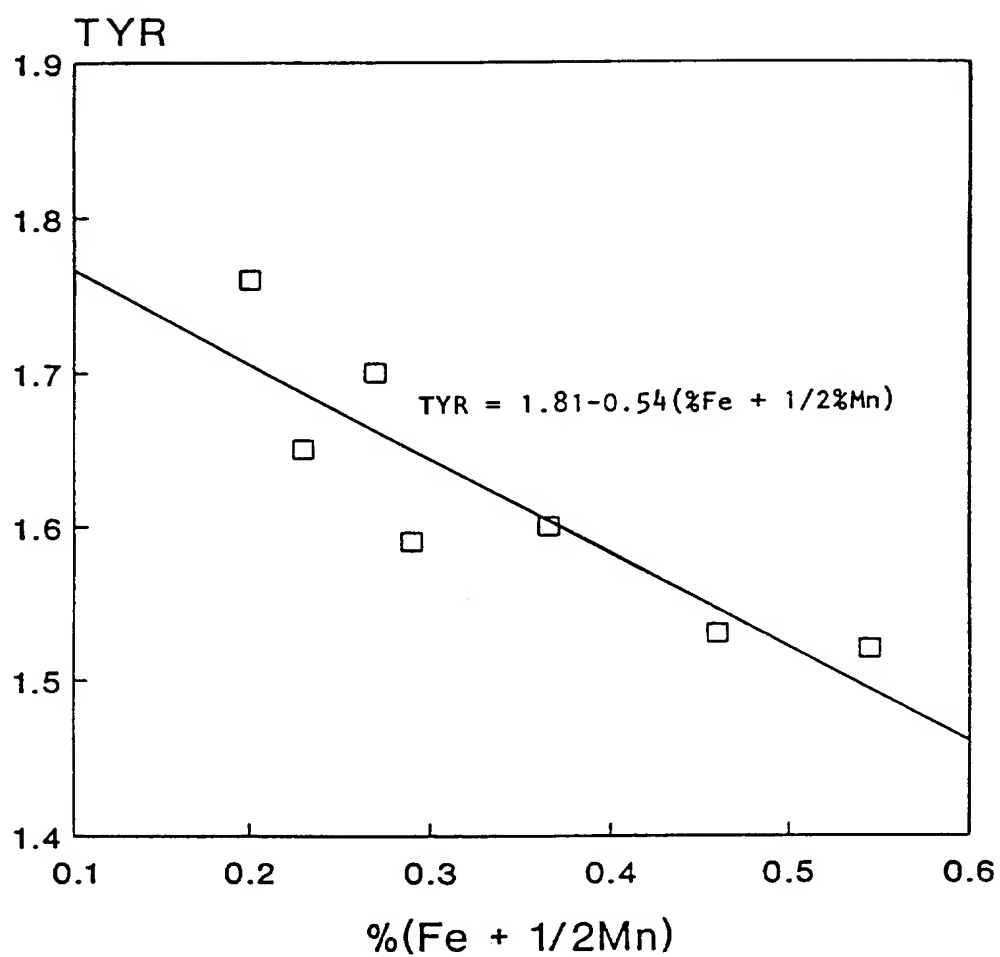


FIG. 5

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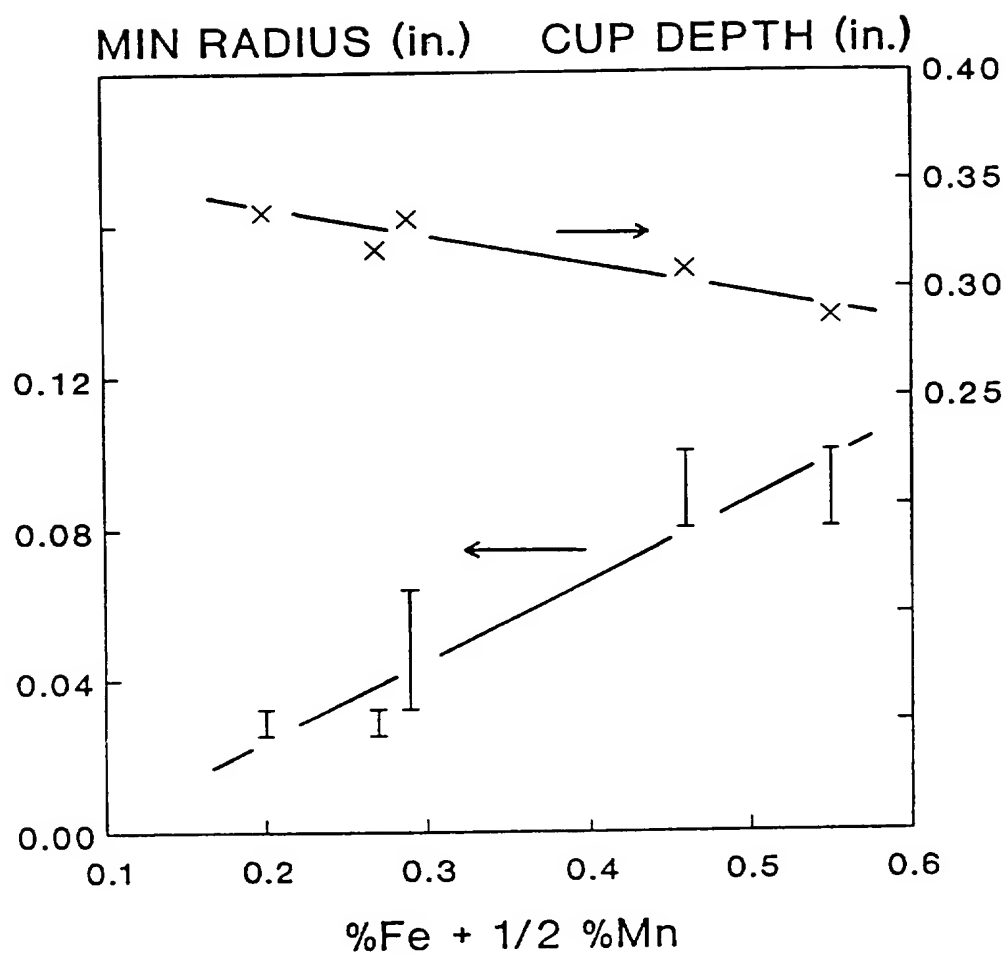


FIG. 6

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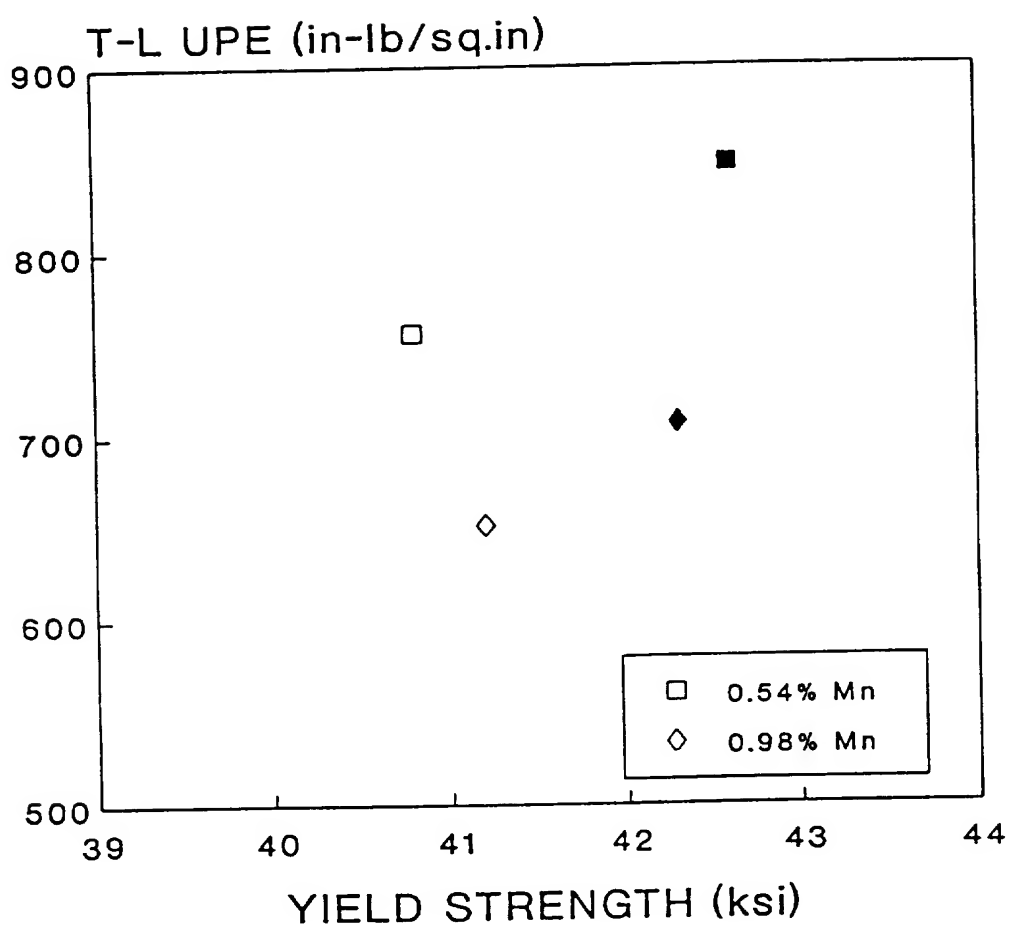
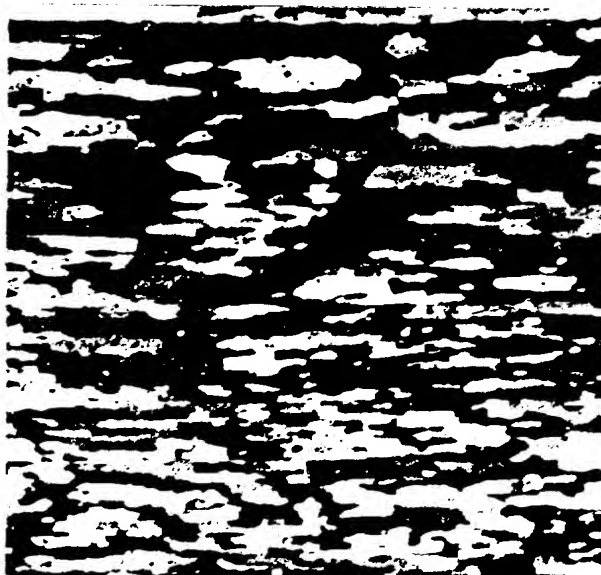
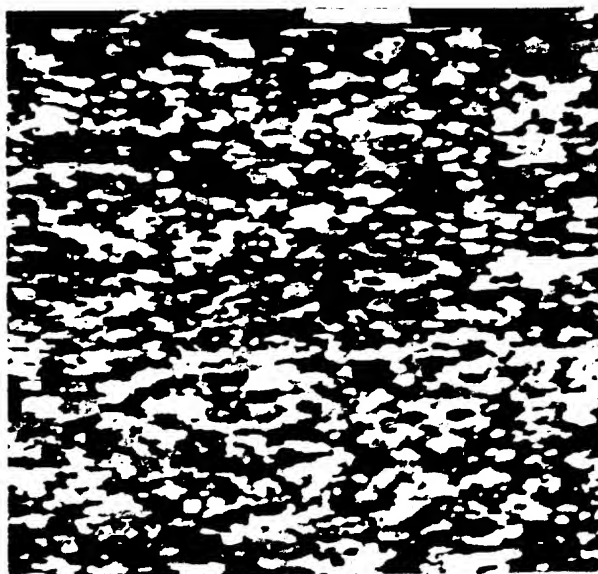


FIG. 7



*FIG. 8a*



*FIG. 8b*

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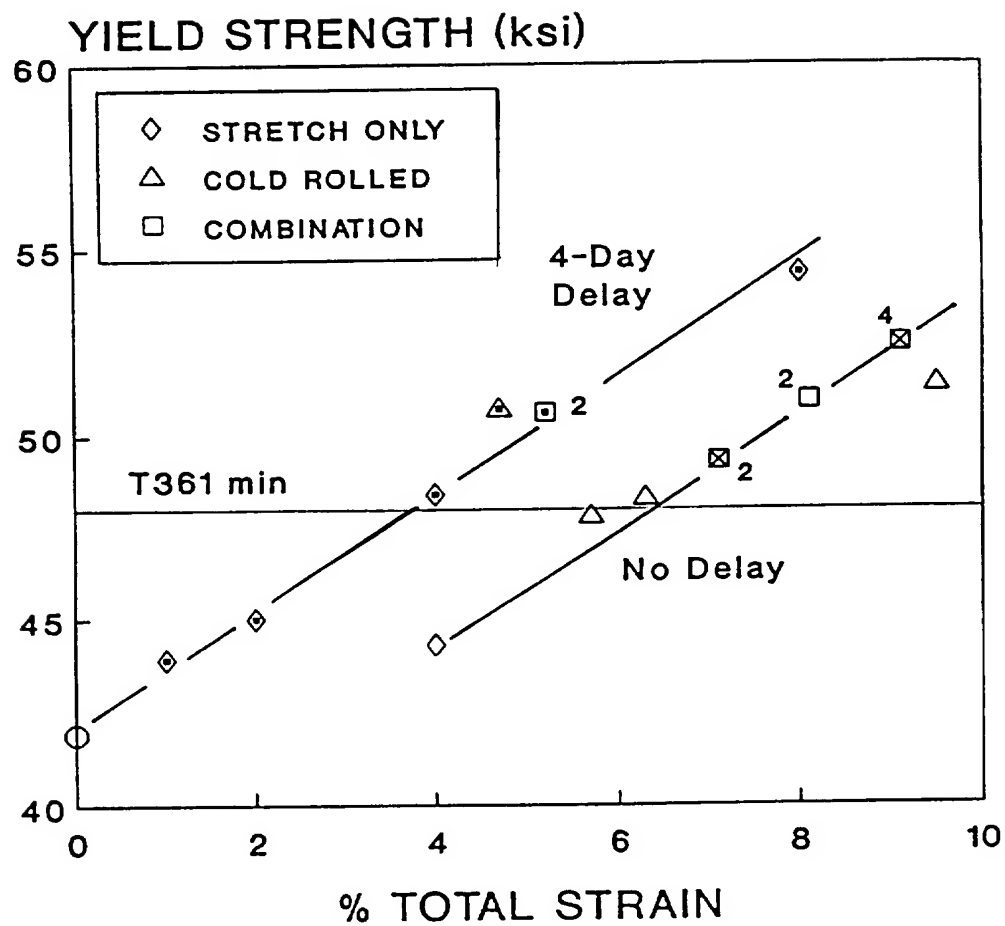


*FIG. 9a*



*FIG. 9b*

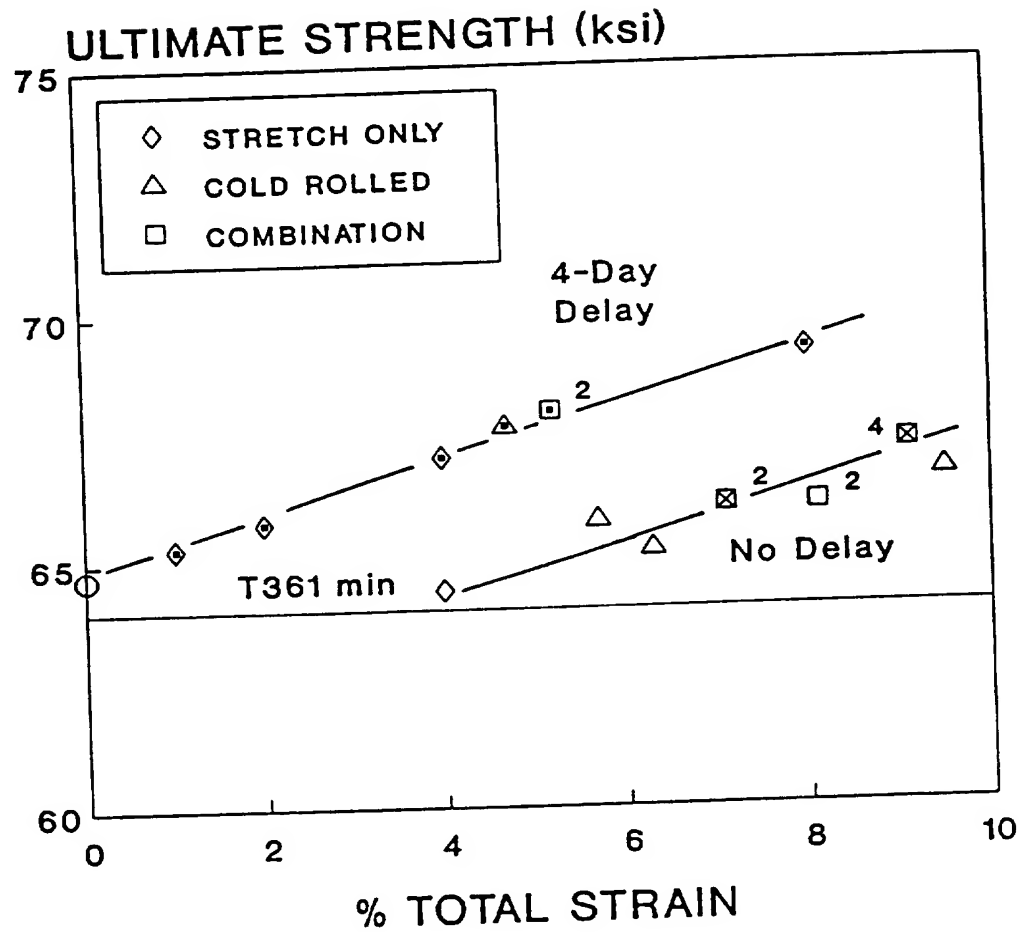
10/17



x - Stretched after 4 days

FIG. 10

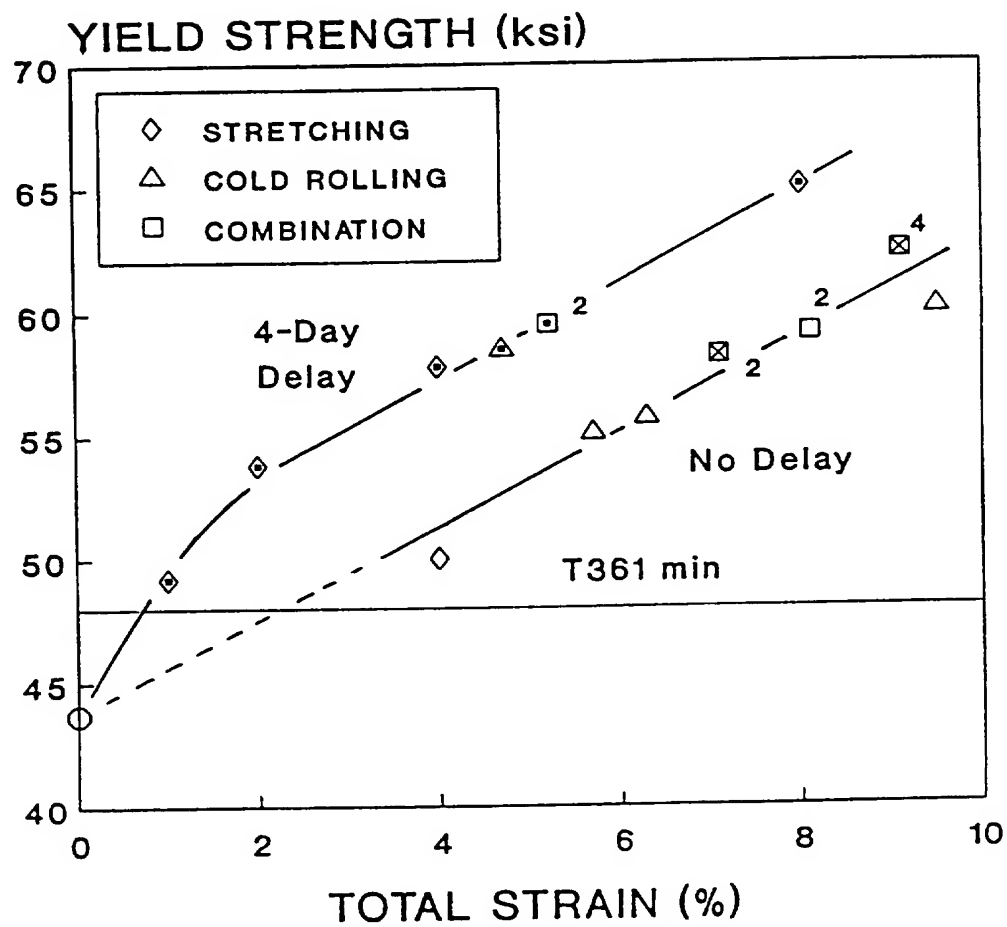
11/17



⊠ - Stretched after 4 days

FIG. 11

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⊠ - Stretched after 4 days

FIG. 12



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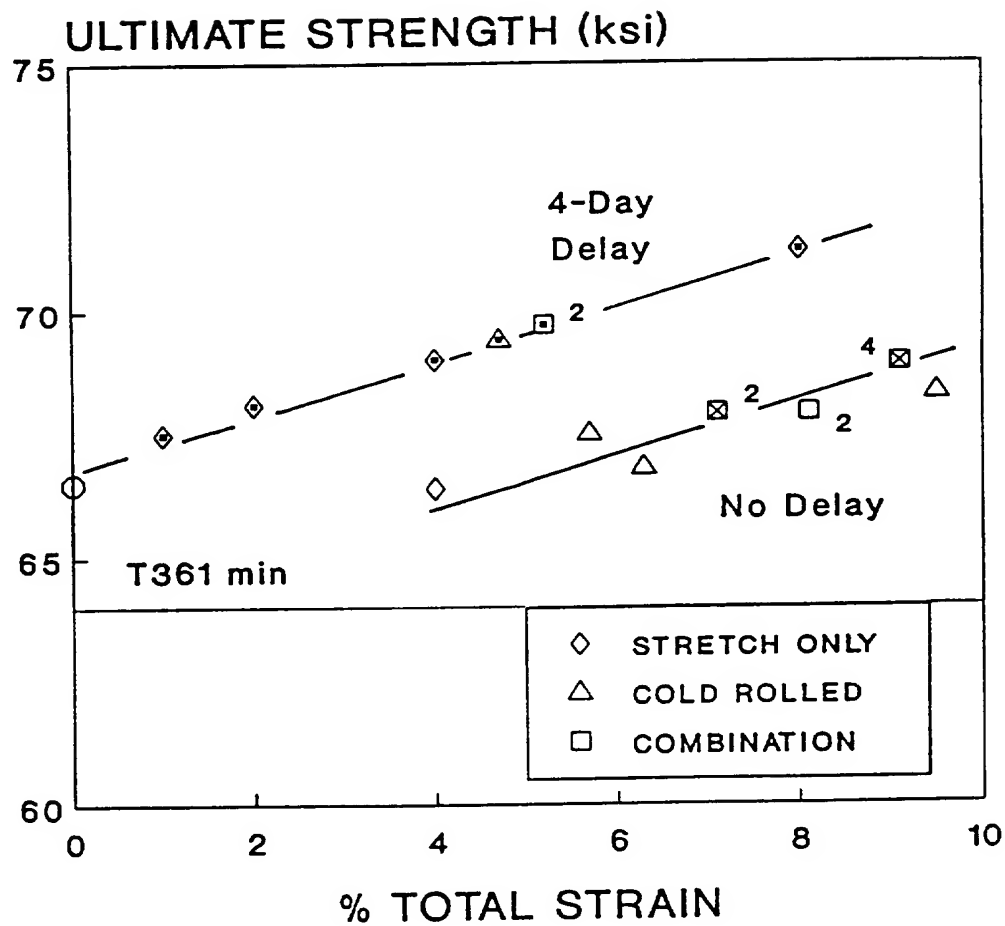
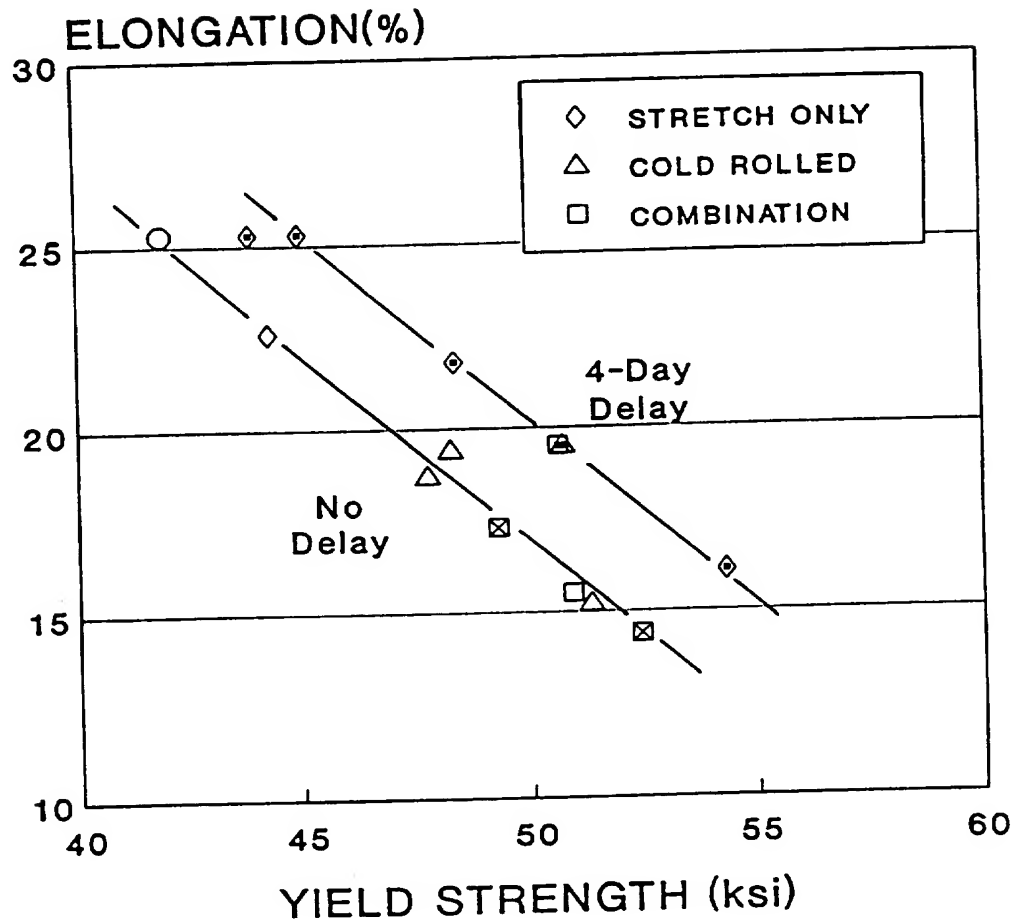


FIG. 13

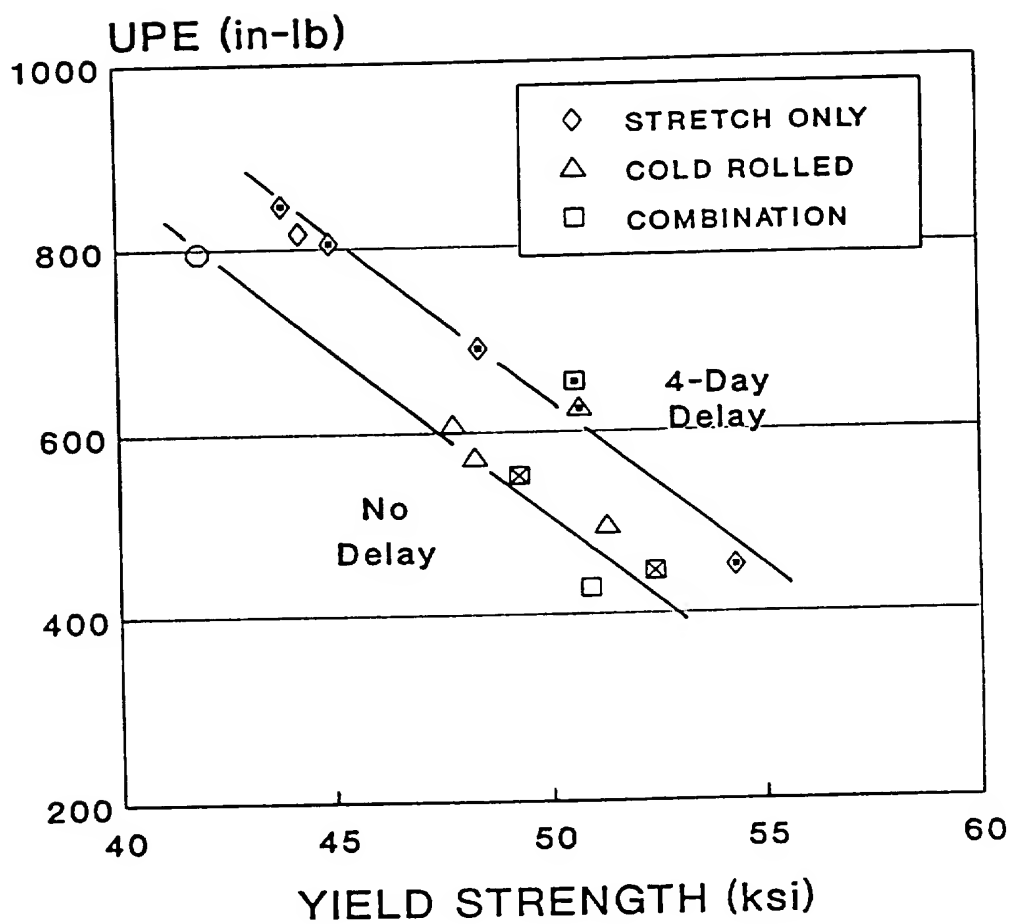
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⊠ - Stretched after 4 days

FIG. 14

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⊠ - Stretched after 4 days

FIG. 15

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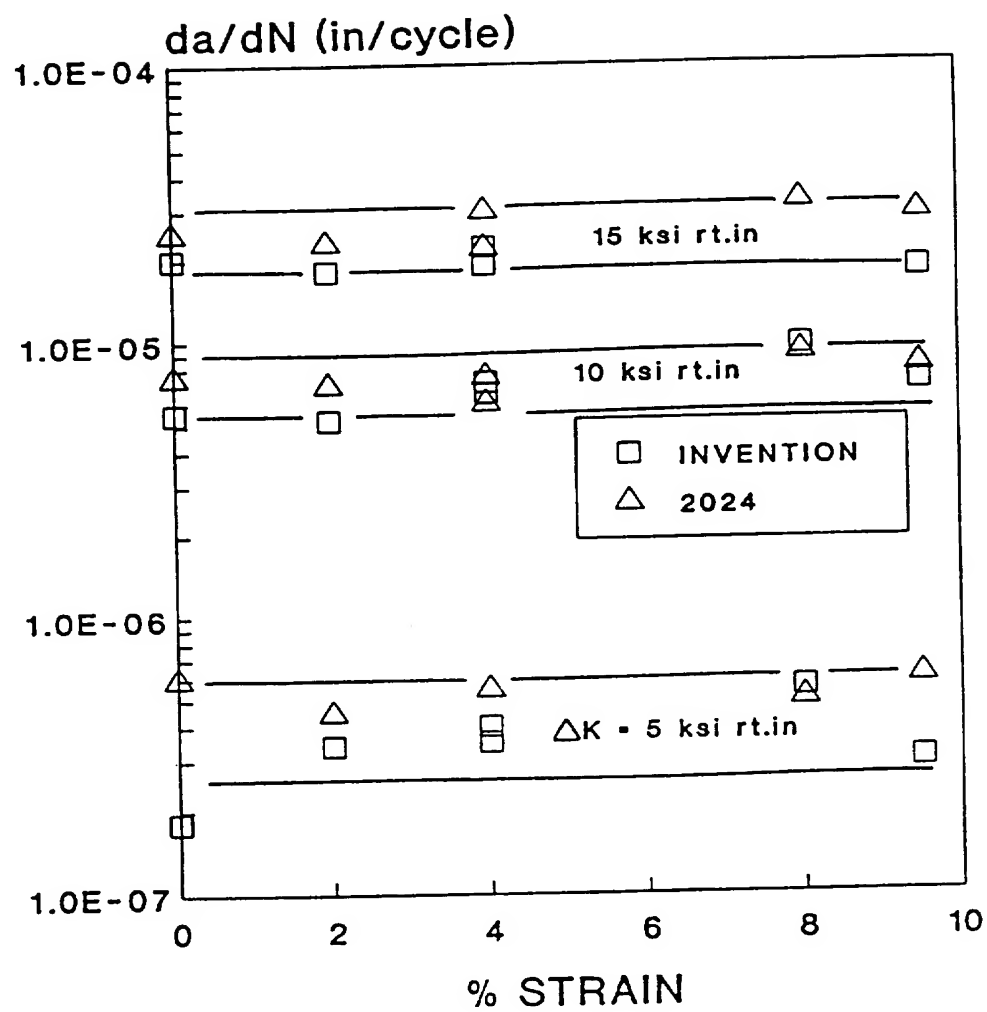


FIG. 16

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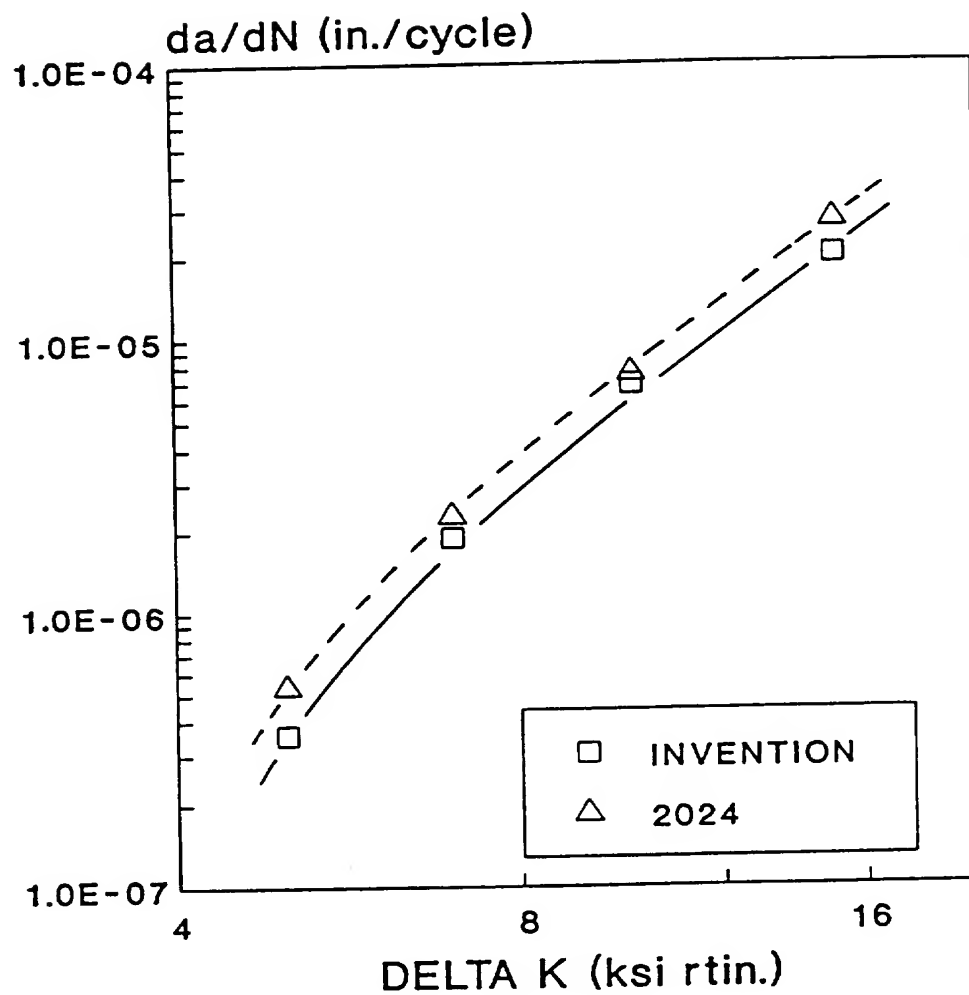


FIG. 17

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/03390

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C22F 1/04

US CL : Please See Extra Sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 148/693, 700, 439; 420/533, 534, 535, 542, 543, 544, 546, 547, 553

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
None

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
None

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y ----- A	US, A, 5,213,639 (COLVIN ET AL) 25 May 1993, col. 3, line 9 to col. 4, line 35, col. 5, line 9 to col. 8, line 44, and col. 8, line 65 to col. 9, line 16.	1-5, 7-16, 18-22 ----- 6, 17



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
*A* document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
*E* earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*Z* document member of the same patent family
*O* document referring to an oral disclosure, use, exhibition or other means	
*P* document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

27 JUNE 1996

Date of mailing of the international search report

12 JUL 1996

Name and mailing address of the ISA/US  
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# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/03390

A. CLASSIFICATION OF SUBJECT MATTER:  
US CL :

148/693, 700, 439; 420/533, 534, 535, 542, 543, 544, 546, 547, 553